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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6:		(11) International Publication Number:	WO 95/06700
C10M 129/70, 129/74, 143/06	A1	(43) International Publication Date:	9 March 1995 (09.03.95)
 (21) International Application Number: PCT/US (22) International Filing Date: 22 August 1994 ((30) Priority Data: 08/114,716 31 August 1993 (31.08.93) (71) Applicant: HENKEL CORPORATION [US/US]; S 140 Germantown Pike, Plymouth Meeting, PA 19 (72) Inventor: ZEHLER, Eugene, R.; 7449 Cinnamo Drive, West Chester, OH 45069 (US). (74) Agent: DRACH, John, E.; Henkel Corporation, Suite Germantown Pike, Plymouth Meeting, PA 19462 (22.08.5 Suite 1 462 (U	CN, CZ, DE, DK, ES, FI, GB, KR, KZ, LK, LT, LU, LV, MD NZ, PL, PT, RO, RU, SD, SE, VN, European patent (AT, BE, GR, IE, IT, LU, MC, NL, PT, CF, CG, CI, CM, GA, GN, MI ARIPO patent (KE, MW, SD). Published With international search reported	GE, HU, JP, KE, KG, KP, MG, MN, MW, NL, NO. SI, SK, TJ, TT, UA, UZ CH, DE, DK, ES, FR, GB SE), OAPI patent (BF, BJ L, MR, NE, SN, TD, TG)
(54) Title: EXTREME PRESSURE LUBRICANT			

(57) Abstract

An extreme pressure industrial gear lubricant having enhanced oxidative and thermal stability, lower pour point, and higher viscosity index relative to petroleum-based lubricants is comprised of a poly- α -olefin, a polyol ester, a polybutene oligomer, an antioxidant, and an extreme pressure additive.

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EXTREME PRESSURE LUBRICANT BACKGROUND OF THE INVENTION

1. Field of the Invention

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This invention relates to industrial extreme pressure gear lubricants having enhanced oxidative and thermal stability.

2. Description of the Related Art

Industrial extreme pressure gear lubricants are used in practically every aspect of the manufacturing and processing industry. Industrial gear lubricants are most frequently used in such components as reduction gears, drive units, screws, slides, chains, and the like. Of the three recognized types of industrial gear lubricants, the most important are the extreme pressure or EP gear oils. In addition to protecting metal parts from corrosion and thermal and oxidative deterioration, EP gear oils must also provide protection against scoring and other types of mechanical distress. EP gear oils are based on petroleumbased or synthetic materials. The petroleum-based EP oils are usually comprised of a petroleum-based material such as mineral oil, an antioxidant, an antifoam, a corrosion inhibitor, and one or more EP additives. In synthetic gear oils, the petroleum-based material is replaced with such synthetic substances as hydrocarbons, esters, polyglycols,

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phosphate esters, silicones, silicate esters, polyphenyl ethers, and halogenated hydrocarbons depending upon the particular application. It is well known in the art that synthetic gear lubricants tend to last longer, exhibit better high temperature stability, have higher viscosity indexes, and usually have lower pour points than petroleumbased lubricants. Because industrial gear horsepower ratings have increased fourfold over the last 15 years, gear oils are subjected to increasingly higher temperatures which causes a correspondingly shorter service life due to thermal and oxidative degradation. Thus there is always a need for industrial EP gear oils which can provide enhanced oxidative and thermal stability. The compositions according to the invention are industrial gear lubricants having improved high temperature performance, high temperature stability, and cleanliness.

SUMMARY OF THE INVENTION

It has been discovered surprisingly that a composition which is comprised of: (a) a poly- α -olefin which has a viscosity of from 4 centistokes to 100 centistokes @ 100°C; (b) a polyol ester made by reacting a monocarboxylic acid having from 5 to 18 carbon atoms and a polyol which has at least 3 alcohol functionalities; (c) a polybutene having a molecular weight of from about 700 to about 2500 Daltons; (d) an antioxidant; (e) a sulfur/phosphorus type extreme pressure additive having a specific gravity @ 15.6°C equal to 1.022; a viscosity in centistokes @ 100°C equal to 14.3; color according to ASTM D 1500 equal to 4.0; % boron by weight equal to 0.36; % nitrogen by weight equal to 1.21; % phosphorus by weight equal to 1.61; % sulfur by weight equal to 19.6 affords an extreme pressure industrial gear lubricant having enhanced oxidative and thermal stability, and higher viscosity index relative to petroleum-based lubricants and other polyol ester-based lubricants.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Other than in the operating examples, or where otherwise indicated, all numbers expressing quantities of

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ingredients or reaction conditions used herein are to be understood as modified in all instances by the term

The poly- α -olefins which can be used in "about". compositions according to the invention are those which have a viscosity in the range of from about 4 centistokes to about 100 centistokes @ 100°C. Preferred poly- α -olefins are those which have a viscosity in the range of from about 4 centistokes to about 10 centistokes @ 100°C. The amount of poly- α -olefin which can be used in the compositions according to the invention can range from 20% by weight to 90% by weight and will preferably be in the 25% by weight to 55% by weight range.

The polyol esters which can be used in the composition according to the invention are those which can be made by esterifying monocarboxylic acids having from 5 to 18 carbon with a polyol having at least 3 functionalities examples of which include but are not limited to such polyols as neopentyl glycol (2,2-dimethyltrimethylolethane (hydroxymethyl)-1,3-propanediol], trimethylolpropane [2-1,3-propanediol), ethyl-2-(hydroxymethyl)-1,3-propanediol], pentaerythritol, glycerine, The preferred polyol esters are esters of dipentaerythritol, pelargonic acid. The most preferred polyol ester according triglycerine. to the invention is trimethylolpropane tripelargonate. The molecular weight of the polyol esters which can be used can range from 270 to 1,900 with those having a molecular weight of from 480 to 1,400 being preferred. The amount of polyol ester which can be used in the compositions according to the invention can range from 5% by weight to 50% by weight and will preferably be in the 10% by weight

The polybutenes which can be used in the compositions to 30% by weight range. according to the invention are polybutene oligomers having a molecular weight in the range of from 700 to 2500 Daltons with the preferred polybutene having a molecular weight in

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the range of from 1000 to 1500 Daltons. The amount of polybutene which can be used in the compositions according to the invention can range from 1% by weight to 70% by weight and will preferably be in the 20% by weight to 60%

The antioxidants which can be used in the compositions by weight range. according to the invention are substituted diarylamines, phenothiazines, hindered phenols, or the like. Preferred antioxidants include thiodiethylene bis-(3,5-di-tert-butyl-4-hydroxy) hydrocinnamate, available commercially as Irganox® L 115, a trademark product of Ciba-Geigy and a mixture of dioctyl- and dibutyldiphenylamines, available commercially as Irganox® L 57, a trademark product of Ciba-Geigy and combinations of thiodiethylene bis-(3,5-di-tertbutyl-4-hydroxy) hydrocinnamate and a mixture of dioctyland dibutyldiphenylamines. The amount of antioxidant which can be used in the compositions according to the invention can range from 0.05% by weight to 1.0% by weight and will preferably be in the 0.2% by weight to 0.8% by weight range.

The extreme pressure additive which can be used in the compositions according to the invention are thermally stable, sulfur/phosphorus type EP additives such as Lubrizol® 5045 industrial gear oil additive and the like. A typical EP additive will have the following physical properties: (1) specific gravity @ 15.6°C equal to 1.022; (2) viscosity in centistokes @ 100°C equal to 14.3; (3) color, ASTM D 1500 equal to 4.0; (5) % boron by weight equal to 0.36; (6) % nitrogen by weight equal to 1.21; (7) % phosphorus by weight equal to 1.61; (8) % sulfur by weight equal to 19.6. The amount of extreme pressure additive which can be used in the compositions according to the invention can range from 1% by weight to 3% by weight and will preferably be in the 1.5% by weight to 2.5% by

The lubricant compositions according to the invention weight range. are typically made by thoroughly mixing all the components

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together with the aid of conventional mixing equipment while supplying such heating as necessary to maintain

The following examples are meant to illustrate but not fluidity of the mixture. limit the invention.

Lubricant compositions according to the invention were prepared having the compositions, expressed as weight %, set forth in Table 1.

set forth	in Table	TABL	E 1		
			С	D	E
	A	В		34.6	29.1
	52.1	46.1	40.1	12.0	12.0
PAE1		12.0	12.0		56.5
PE2	12.0		45.5	51.0	0.20
PB3	33.5	39.5	0.20	0.20	1
	0.20	0.20	0.20	0.20	0.20
Anti-ox4		0.20		2.00	2.00
Anti-ox5	1 0.20	2.00	2.00 fin having a e 100°C.	mole. Wt of a	pprox. 530
EP6	2.00	a poly-a-ole	fin having a	шо	
1- E	meryo soco,	ty of 6 Cst	e 100°C. Tropane tripela	rgonate	ight in the

and a viscosity of 6 Cst @ 100°C.

2- Emeryw 2934; trimethytolpropane tripelargonate
3- Indopol® H-300; a polybutene having a molecular weight in the range of from 1000 to 1500 Daltons; a trademark product of

4- Irganox® L-115

The data presented in Table 2 show the viscosity 5- Irganox® L-57 index, pour point, viscosity increase & precipitation number as determined under the USS S-200 Oxidation Stability Test protocol for each of the lubricant compositions of Table 1. MARLE 2

mpositions of Table	TABLE	2		E
A VI1 124	B 124 -35°F <4% Trace	C 124 -30°F <4% Trace	D 124 -25°F <4% Trace	124 -15°F <4% Trace

2- rour Form.
3- % Viscosity Increase @ 210°F in USS S-200 Oxidation Stability
Test 1- Viscosity Index

4- Precipitation Number in USS S-200 Oxidation Stability Test

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The data in Table 2 can be compared to the corresponding values for a typical petroleum-based lubricant. For example, the viscosity index for a typical The greater the petroleum-based lubricant is 90-100. viscosity index, the less a particular lubricant's viscosity will change as a function of temperature. The pour point for a typical petroleum-based lubricant is 0°F to +10°F. The lower the pour point the better a particular lubricant will flow at lower temperatures and therefore, the better it will lubricate. The % viscosity increase under the USS S-200 protocol must be 6 or less and is typically 5 for acceptable petroleum-based lubricants. The precipitation number under the USS S-200 protocol, which is a measure of the sludge formation, must be less than 0.10 and is typically 0.05 for petroleum-based lubricants.

Effect of Lubricant Composition on Coking Tendency

lubricant compositions was determined by the Panel Coke Test and is given in Table 3. The coking tendency measures the likelihood that a particular lubricant will form solid decomposition products when in contact with surfaces at elevated temperatures and is measured by the weight gain of a panel, in milligrams, after the test. The larger the weight gain, the greater the tendency of a particular lubricant to decompose under the test conditions.

Lubricant A is a composition according to the invention and was comprised, in weight % of: (a) 46.8% 12.0% a mixture of monodipentaerythritol ester of iso-C5 and n-C9 carboxylic acids having a viscosity of about 5.0 Cst @ 100°C; 39.0% Indopol® H-300; 0.20% Irganox® L-115; 0.20% Irganox® L-57; and 1.8% Lubrizol® 5045. Lubricant A had a viscosity of 220 Cst @ 40°C. Lubricant B was a standard ISO 220 petroleum-based industrial gear lubricant having 1.8% Lubrizol® 5045 and Lubricant C was a standard ISO 220 petroleum-based gear

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lubricant containing a typical EP additive other than The data show that Lubrizol® 5045 is extremely effective in reducing the amount of solid Lubrizol® 5045. decomposition products when in contact with surfaces at elevated temperatures as measured by the Panel Coke Test. The data also shows that Lubricant A, a composition according to the invention, exhibits a reduced tendency to form solid decomposition products relative to ISO 220 petroleum-based industrial gear lubricant, containing Lubrizol® 5045. An EP additive such as Lubrizol® 5045 is most effective in reducing the amount of high temperature decomposition of gear lubricants when used in combination with C_{5-18} monocarboxylic acid esters of a polyol having at least 3 alcohol functionalities.

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18 functional:	ffres.	
alcohol functional		
	Lubric	ant
	A B	147.6
Panel Wt. Gain (mg)	6.4 8.4	
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EXAMPLE 4 Coking Tendency Test Method

The method is based on Federal Test Standard 791B The apparatus used in the test can be a Roxanna Model C Panel Coker, A Falex Panel Coker, or Method 3462.

An aluminum test panel is polished to a dull luster with fine steel wool, washed with petroleum ether, and equivalent. weighed to the nearest 0.10 milligram. About 270 ml of test oil is poured into the coker body. The test panel is placed above the coker body in the sliding panel runway so that the polished surface is exposed to oil from the splasher. The strip heater is placed above the test panel and tightened securely. The test temperature is monitored by a thermocouple which is inserted are into the test panel. Oil is splashed onto the test panel continuously throughout the duration of the test. The tests are performed for a time period of 4 hours at 260°C. When the test period is over, the coker panel is cooled, removed, and washed with several

portions of petroleum ether, and reweighed. The difference in weight of the test panel is reported as coking value.

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What is claimed is:

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- An extreme pressure gear lubricant composition comprising: (a) a poly- α -olefin having a viscosity of from about 4 centistokes to about 100 centistokes @ 100°C; (b) a polyol ester wherein the carboxylic acid component is a monocarboxylic acid having from 5 to 18 carbon atoms and 5 wherein the polyol component is polyol having at least 3 alcohol functionalities; (c) polybutene having a molecular weight of from about 700 to about 2500 Daltons; (d) an antioxidant; (e) a sulfur/phosphorus type extreme pressure additive having a specific gravity @ 15.6°C equal to 1.022; 10 a viscosity in centistokes @ 100°C equal to 14.3; color according to ASTM D 1500 equal to 4.0; % boron by weight equal to 0.36; % nitrogen by weight equal to 1.21; % phosphorus by weight equal to 1.61; % sulfur by weight 15 equal to 19.6.
- The composition of claim 1 wherein said poly- α -olefin has a viscosity of from about 4 to about 10 centistokes @ 100°C. . 20
 - The composition of claim 1 wherein said polybutene is a polybutene oligomer having a molecular weight of from 1,000 to 1,500 Daltons.
 - The composition of claim 1 wherein said antioxidant is selected from the group consisting of: (i) thiodiethylene bis-(3,5-di-tert-butyl-4-hydroxy) hydrocinnamate, (ii) a mixture of dioctyl- and dibutyldiphenylamines and, (iii) a combination of (i) and (ii).
 - The composition of claim 1 wherein said polyol ester is trimethylolpropane tripelargonate.
 - The composition of claim 1 wherein the molecular of said polyol ester is from about 270 to about 1,900 Daltons. 35

- 7. The composition of claim 6 wherein said molecular weight is from about 480 to about 1,400.
- 8. The composition of claim 1 wherein the amount of said polyol ester in said composition is from about 5% by weight to about 50% by weight.
- 9. The composition of claim 8 wherein the amount of said polyol ester in said composition is from about 10% by weight to about 30% by weight.
 - 10. The composition of claim 1 wherein the amount of said poly- α -olefin in said composition is from about 20% by weight to about 90% by weight.
- 15 11. The composition of claim 10 wherein the amount of said poly-α-olefin in said composition is from about 25% by weight to about 55% by weight.
- 12. The composition of claim 1 wherein the amount of said polybutene in said composition is from about 1% by weight to about 70% by weight.
- 13. The composition of claim 12 wherein the amount of said polybutene in said composition is from about 20% by weight to about 60% by weight.
- 14. The composition of claim 1 wherein the amount of said antioxidant in said composition is from about 0.05% by weight to about 1.0% by weight.

- 15. The composition of claim 14 wherein the amount of said antioxidant in said composition is from about 0.2% by weight to about 0.8% by weight.
- 16. The composition of claim 1 wherein the amount of said extreme pressure additive in said composition is from about

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1% by weight to about 3% by weight.

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- 17. The composition of claim 1 wherein the amount of said extreme pressure additive in said composition is from about 1.5% by weight to about 2.5% by weight.
- extreme pressure gear lubricant composition comprising: (a) from about 20% by weight to about 90% by weight of a poly- α -olefin having a viscosity of from about 4 centistokes to about 100 centistokes @ 100°C; (b) from 10 about 5% by weight to about 50% by weight of a polyol ester wherein the carboxylic acid component is a monocarboxylic acid having from 5 to 18 carbon atoms and wherein the polyol component is polyol having at least 3 alcohol functionalities; (c) from about 1% by weight to about 70% 15 by weight of a polybutene having a molecular weight of from about 700 to about 2500 Daltons; (d) from about 0.05% by weight to about 1.0% by weight of an antioxidant; (e) from 1% by weight to about 3% by weight of a sulfur/phosphorus type extreme pressure additive having a 20 specific gravity @ 15.6°C equal to 1.022; a viscosity in centistokes @ 100°C equal to 14.3; color according to ASTM D 1500 equal to 4.0; % boron by weight equal to 0.36; % nitrogen by weight equal to 1.21; % phosphorus by weight equal to 1.61; % sulfur by weight equal to 19.6. 25
 - 19. The composition of claim 18 wherein said is selected from the group consisting of: (i) thiodiethylene bis-(3,5-di-tert-butyl-4-hydroxy) hydrocinnamate, (ii) a mixture of dioctyl- and dibutyldiphenylamines and, (iii) a combination of (i) and (ii).
 - 20. The composition of claim 18 wherein said polyol ester is trimethylolpropane tripelargonate.
 - 21. The composition of claim 18 wherein the molecular of said polyol ester is from about 270 to about 1,900 Daltons.

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22. The composition of claim 21 wherein said molecular weight is from about 480 to about 1,400.

- 23. The composition of claim 18 wherein the amount of said polyol ester in said composition is from about 10% by weight to about 30% by weight.
- 24. The composition of claim 18 wherein the amount of said poly- α -olefin in said composition is from about 25% by weight to about 55% by weight.
 - 25. The composition of claim 18 wherein the amount of said polybutene in said composition is from about 20% by weight to about 60% by weight.

26. The composition of claim 18 wherein the amount of said extreme pressure additive in said composition is from about 1.5% by weight to about 2.5% by weight.

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20 📉 27. An extreme pressure gear lubricant composition comprising: (a) from about 20% by weight to about 90% by weight of a poly- α -olefin having a viscosity of from about 4 centistokes to about 10 centistokes @ 100°C; (b) from by weight to about 50% by weight about 5₺ trimethylolpropane tripelargonate; (c) from about 1% by 25 weight to about 70% by weight of a polybutene having a molecular weight of from about 700 to about 2500 Daltons; (d) from about 0.05% by weight to about 1.0% by weight of an antioxidant is selected from the group consisting of: thiodiethylene bis-(3,5-di-tert-butyl-4-hydroxy) 30 (i) hydrocinnamate, (ii) a mixture of dioctyldibutyldiphenylamines and, (iii) a combination of (i) and (ii); (e) from about 1% by weight to about 3% by weight of a sulfur/phosphorus type extreme pressure additive having a specific gravity @ 15.6°C equal to 1.022; a viscosity in 35 centistokes @ 100°C equal to 14.3; color according to ASTM

D 1500 equal to 4.0; % boron by weight equal to 0.36; %

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nitrogen by weight equal to 1.21; % phosphorus by weight equal to 1.61; % sulfur by weight equal to 19.6.

extreme pressure gear lubricant composition comprising: (a) from about 20% by weight to about 90% by weight of a poly-α-olefin having a viscosity of from about 4 centistokes to about 10 centistokes @ 100°C; (b) from about 5% by weight to about 50% by weight of a mixture of mono- and dipentaerythritol esters of iso-C5 and n-C9 carboxylic acids having a viscosity of about 5.0 Cst @ 100°C; (c) from about 1% by weight to about 70% by weight of a polybutene having a molecular weight of from about 700 to about 2500 Daltons; (d) from about 0.05% by weight to about 1.0% by weight of an antioxidant is selected from the group consisting of: (i) thiodiethylene bis-(3,5-di-tertbutyl-4-hydroxy) hydrocinnamate, (ii) a mixture of dioctyland dibutyldiphenylamines and, (iii) a combination of (i) and (ii); (e) from about 1% by weight to about 3% by weight of a sulfur/phosphorus type extreme pressure additive having a specific gravity @ 15.6°C equal to 1.022; a viscosity in centistokes @ 100°C equal to 14.3; color according to ASTM D 1500 equal to 4.0; % boron by weight equal to 0.36; % nitrogen by weight equal to 1.21; % phosphorus by weight equal to 1.61; % sulfur by weight equal to 19.6.

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INTERNATIONAL SEARCH REPORT

International application No. PCT/US94/09134

A. CL	ASSIFICATION OF SUBJECT MATTER				
IPC(6) :C10M 129/70, 129/74, 143/06					
US CL	US CL :252/42.7, 56 S				
According	According to International Patent Classification (IPC) or to both national classification and IPC				
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C. DOC	CUMENTS CONSIDERED TO BE RELEVANT				
	<u> </u>				
Category*	Citation of document, with indication, where	appropriate, of the relevant passages	Relevant to claim No.		
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INTERNATIONAL SEARCH REPORT

International application No. PCT/US94/09134

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
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	EP, 0,119,069, (Loveless et al.) 19 September 1984, page 8, lines 5-13; page 11, lines 6-12; claims 1 and 2.	1-2,5,8-11,14- 18,20,23-24,26- 28
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